UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
REGION II

DATE: **June 1, 2006**

SUBJECT: Chemical Data Assessment - Sampling and Testing of Material Proposed for Dredging from the San Juan Harbor Section 103, Puerto Rico.

FROM: Carol L. Lynes, Environmental Scientist
Monitoring and Assessment Branch

TO: Mark Reiss, Environmental Scientist
Dredged Material Management Team

As per your request, I have completed our assessment of the chemical data summaries produced for the San Juan Harbor Dredging Project. Specific review criteria and comments are included in the attached chemical data assessment report.

The assessment of the chemical data associated with this project concludes that the data are "acceptable", "not acceptable", and that a determination of acceptability could not be made. Details of the data acceptability determinations and the inability to determine acceptability are provided in the attached report narrative and tables. Where a determination could not be made from the quality control criteria the data users must determine the usability of these data.

If you have any questions, please feel free to contact me at 732-321-6760 or lynes.carol@epa.gov.

Attachments

Cc. J. Ferretti, DESA-LAB

SUMMARY OF FINDINGS AND TECHNICAL RECOMMENDATIONS - CHEMICAL TESTING

Project(s): Dredge Material Testing for Proposed Ocean Disposal - San Juan Harbor Section 103
Chemical Testing Laboratories: PPB Environmental Service with sub-contracted analytical services to Columbia Analytical Services

Chemical testing was conducted on site water, elutriate, sediment and tissue matrices. The site water/elutriate analysis consisted of Pesticides, PCB Congeners, and Metals; the sediment analysis consisted of PAH Compounds, Pesticides, PCB Congeners, and Metals; and the tissue analysis consisted of PAH Compounds, Pesticides, PCB Congeners, and Metals. Tissue, site water, and elutriate summary data, including quality control results, were reviewed for compliance with the established testing requirements specified in the Regional Guidance For Performing Tests On Dredged Material Proposed For Ocean Disposal (USEPA/USACE-NYD, 1992 with modifications), hereafter referred to as the Regional Testing Manual (RTM).

The assessment and conclusions on data acceptability are outlined in the following tables. Three tables - "Tissue", "Site Water", and "Elutriate" - are dedicated for each of the chemical testing groups, i.e., PAH Compounds, Pesticides/PCB Congeners, and Metals.

The acceptability of the data for each chemical group and matrix is categorized as "acceptable", "acceptable with condition", "unacceptable", and/or "not determined". It is our opinion that the data with an "acceptable" or "acceptable with condition" may be used; however, if indicated, please note the particular condition. The category "not determined" is used if insufficient data were provided.

Data Reporting Comments:
Samples, including the QC samples, are not separated and organized into analytical batches, thus requiring EPA to utilize extraction and analysis dates and the extraction lot numbers, in order to make the determination as to which samples were batched together. This is not good laboratory reporting practices and will not be accepted in future packages. It is the responsibility of PPB Environmental Services to ensure that the analytical data packages submitted to EPA are well organized and include all appropriate QC samples.

Laboratory Analysis Comments:
The relationship of the MRL to the MDL is not clear. It is not understood why the MRL and MDL change from sample to sample within the same extraction/analytical batch.

The MRL exceeds the program action level for the pesticides Endosulfan I, Endosulfan II and Endosulfan Sulfate in lab code numbers K0503972-005, K0503972-011, K0503972-013, K0503972-015, K0503972-031.

The SRM samples submitted for the Pesticides and PCBs in tissue were not extracted or analyzed with the associated samples in the analytical batch. Therefore, it is not representative of the batch and is not acceptable. It is indicated on the SRM sample QA/QC Report for PCBs that the SRM sample was extracted on 10/14/05 and was analyzed on 10/24/05. However, the signature of approval is dated 4/6/06. It is indicated on the SRM sample QA/QC Report for Pesticides that the SRM sample was extracted on 10/14/05 and was analyzed on 10/25/05. However, the signature of approval is not dated.

Some of the matrix spike samples did not contain all of the required analytes as present in the RTM. Matrix Spike samples must contain all of the analytes of interest. This program does not accept a subset of the analytes of interest such as in the CERCLA or RCRA programs.

The tissue samples for metals analysis, including the QC samples, are not separated and organized into analytical batches, thus, from the information provided the analytical batches along with the associated QC samples could not be determined.

The tables below provided additional information specific to the problems encountered during the data review.

PAH COMPOUNDS	TISSUE
MDL VERIFICATION STUDY (PAHs 4 ng/g wet wt.)	MDL verifications were not performed with the project. The lab submitted past MDL studies for the parameters of interest in the matrix of interest. The MDL studies submitted were reviewed and found to be acceptable.
HOLDING TIME	Acceptable
STANDARD REFERENCE MATERIALS (SRM) SAMPLE(S)	SRM % Difference: (≤ 30%D of cv on avg for analytes > 10x MDL; ≤ 35% of all recoveries within a set of analytes > 30%D; No SRM recovery > 50%D) *N. virens* : An SRM sample was not analyzed. Analyzing an SRM after the fact and not with the analytical batch is not acceptable. The laboratory did analyze a LCS in tissue. EPA used the LCS in place of an SRM sample. Although the concentrations in the LCS are not certified we are willing to accept the LCS for this project. This is not an acceptable laboratory practice for this program. An SRM sample will be expected in all future data packages. The results of the LCS are within the established criteria. *M. nasuta*: Same comment as above.
GENERAL QUALITY CONTROL SAMPLES (Replicate Samples; Matrix Spikes; Surrogates; Method Blanks)	Triplicate %RSD:(≤30% for analytes > 10x the MDL, ≤ 35% RSDs can be > 30%; No RSD > 70% for analytes > 10x MDL) *N. virens*: Triplicate analyses were not performed as required in the RTM. Analyzing triplicate samples after the fact and not with the analytical batch is not acceptable. *M. nasuta*: Same comment as above. Matrix Spike % Recovery: (50 - 120% for at least 80% of the analytes) *N. virens*: The recoveries are within the established criterion. *M. nasuta*: The recoveries are within the established criterion. Surrogate % Recovery: (30 - 150%) *N. virens*: The recoveries are within the established criterion. *M. nasuta*: The recoveries are within the established criterion. Method Blank Results: (No analyte > 3x the target MDL) *N. virens*: The results are within the established criterion. *M. nasuta*: The results are within the established criterion.
DATA ACCEPTABILITY Acceptable; Acceptable w/Condition; Unacceptable; Not Determined	*N. virens*: Acceptable *M. nasuta*: Acceptable

PESTICIDES and PCB CONGENERS	TISSUE
MDL VERIFICATION STUDY (0.4 ng/g wet)	MDL verifications were not performed with the project. The lab submitted past MDL studies for the parameters of interest in the matrix of interest. The MDL studies submitted were reviewed and found to be acceptable.
HOLDING TIME	Acceptable
STANDARD REFERENCE MATERIALS (SRM) SAMPLE(S)	SRM %Difference: (≤ 30%D of cv on avg for analytes > 10x MDL; ≤35% of all recoveries within a set of analytes > 30%D; No SRM recovery > 50%) Pesticides: One NIST SRM (1945) was analyzed for PCBs in Tissue for a total of 36 samples. The requirement is one SRM per 20 samples. The % Difference from the certified true value was not calculated by the lab. The SRM sample was not extracted or analyzed with the associated samples in the analytical batch. Therefore, it is not representative of the batch and is not acceptable. PCBs: One NIST SRM (1974b) was analyzed for PCBs in Tissue for a total of 36 samples. The requirement is one SRM per 20 samples. The % Difference from the certified true value was not calculated by the lab. The SRM sample was not extracted or analyzed with the associated samples in the analytical batch. Therefore, it is not representative of the batch and is not acceptable.

PESTICIDES and PCB CONGENERS	TISSUE
GENERAL QUALITY CONTROL SAMPLES (Replicate Samples; Matrix Spikes; Surrogates; Method Blanks)	Triplicate %RSD: (≤30% for analytes > 10x the MDL, ≤ 35% RSDs can be >30%, No RSD > 70% for analytes > 10x MDL) *M. nasuta* Pesticides/PCBs: Triplicate analyses were not performed as required in the RTM. The lab proposal of analyzing triplicate samples after the fact and not with the analytical batch is not acceptable. *N. virens* Pesticides/PCBs: Triplicate analyses were not performed as required in the RTM. The lab proposal of analyzing triplicate samples after the fact and not with the analytical batch is not acceptable. Matrix Spike % Recovery: (50 - 120% for at least 80% of the analytes) *M. nasuta* Pesticides: The MS/MSD samples do not include all of the required analytes. Matrix Spike samples must contain all of the analytes of interest. The analytes missing include: 2,4'-DDE; 2,4-DDD; 2,4'-DDT; and Trans-Nonachlor. *M. nasuta* PCBs: The recoveries are within the established criterion. *N. virens* Pesticides: See the comment for *M. nasuta* above. *N. virens* PCBs: The recoveries are within the established criterion. Surrogate % Recovery: (30 - 150%) *M. nasuta* Pesticides: The recoveries are within the established criterion. *M. nasuta* PCBs: The recoveries are within the established criterion. *N. virens* Pesticides: The recoveries are within the established criterion. *N. virens* PCBs: The recoveries are within the established criterion. Method Blank Results: (No analyte > 3x the target MDL) *M. nasuta* Pesticides: The results are within the established criterion. *M. nasuta* PCBs: The results are within the established criterion. *N. virens* Pesticides: The results are within the established criterion. *N. virens* PCBs: The results are within the established criterion.
DATA ACCEPTABILITY Acceptable; Acceptable w/Condition; Unacceptable; Not Determined	*M. nasuta* Pesticides: Not Acceptable *M. nasuta* PCBs: Not Acceptable *N. virens* Pesticides: Not Acceptable *N. virens* PCBs: Not Acceptable

PESTICIDES and PCB CONGENERS	SITE WATER
MDL VERIFICATION STUDY (Pesticides see table 4-3B of RIM; PCBs 0.0005 ug/L)	MDL verifications were not performed.
HOLDING TIME (7 days; elutriate prep ≤ 14 days from collection)	Pesticides: Acceptable PCBs: Acceptable
STANDARD REFERENCE MATERIALS (SRM) SAMPLE(S)	SRM %Difference: (≤ 30%D of cv on avg for analytes > 10x MDL; ≤35% of all recoveries within a set of analytes > 30%D; No SRM recovery > 50%) Pesticides/PCBs: SRM samples were not analyzed. The LCS may be used if it is representative of marine water. There are no supporting data to indicate that the LCS matrix was marine water vs. laboratory grade water. Also, the LCS analyzed does not include all of the required analytes and is missing pesticides (2,4'-DDD, 2,4'-DDE, and 2,4'-DDT) and PCB congeners (49, 128, and 187).
GENERAL QUALITY CONTROL SAMPLES (Replicate Samples; Matrix Spikes; Surrogates; Method Blanks)	Triplicate %RSD: (≤30% for analytes > 10x the MDL, < 35% RSDs can be >30%, No RSD > 70% for analytes > 10x MDL) Pesticides: Sample analysis was not performed in triplicate as required in the RTM. PCBs: Sample analysis was not performed in triplicate as required in the RTM. Matrix Spike % Recovery: (50 - 120% for at least 80% of the analytes) Pesticides: The MS sample does not include all of the required analytes. PCBs: The MS sample does not include all of the required analytes. Surrogate % Recovery: (30 - 150%) Pesticides: The recoveries are within the established criterion. PCBs: The recoveries are within the established criterion. Method Blank Results: (No analyte > 3x the target MDL) Pesticides: The results are within the established criterion. PCBs: The MRL and MDL reported are greater than 3X the target MDL specified in the RTM.
DATA ACCEPTABILITY Acceptable; Acceptable w/Condition; Unacceptable; Not Determined	Pesticides: Not Acceptable. PCBs: Not Acceptable.

PESTICIDES and PCB CONGENERS	ELUTRIATE **(RS-SJH05)**
MDL VERIFICATION STUDY (Pesticides see table 4-3B of RIM; PCBs 0.0005 ug/L)	MDL verifications were not performed.
HOLDING TIME (7 days; elutriate prep ≤ 14 days from collection)	Pesticides: Acceptable PCBs: Acceptable
STANDARD REFERENCE MATERIALS (SRM) SAMPLE(S)	SRM %Difference: (≤ 30%D of cv on avg for analytes > 10x MDL; ≤35% of all recoveries within a set of analytes > 30%D; No SRM recovery > 50%) Pesticides: A SRM sample was not analyzed. The LCS may be used if it is representative of marine water. There are no supporting data to indicate that the LCS matrix was marine water vs. laboratory grade water. The results of the LCS are within the acceptance criteria. PCBs: A SRM sample was not analyzed. The LCS may be used if it is representative of marine water. The LCS data were not included in the package.
GENERAL QUALITY CONTROL SAMPLES (Replicate Samples; Matrix Spikes; Surrogates; Method Blanks)	Triplicate %RSD: (≤30% for analytes > 10x the MDL, < 35% RSDs can be >30%, No RSD > 70% for analytes > 10x MDL) Pesticides: Sample analysis was not performed in triplicate as required in the RTM. PCBs: Sample analysis was not performed in triplicate as required in the RTM. Matrix Spike % Recovery: (50 - 120% for at least 80% of the analytes) Pesticides: The MS sample does not include all of the required analytes. PCBs: The MS sample data were not included in the package. Surrogate % Recovery: (30 - 150%) Pesticides: The recoveries are within the established criterion. PCBs: The recoveries are within the established criterion. Method Blank Results: (No analyte > 3x the target MDL) Pesticides: The results are within the established criterion. PCBs: The MRL and MDL reported are greater than 3X the target MDL specified in the RTM.
DATA ACCEPTABILITY Acceptable; Acceptable w/Condition; Unacceptable; Not Determined	Pesticides: Not Determined (SRM) PCBs: Not Determined (SRM/MS)

PESTICIDES and PCB CONGENERS	ELUTRIATE **(E-SSH05)**
MDL VERIFICATION STUDY (Pesticides see table 4-3B of RIM; PCBs 0.0005 ug/L)	MDL verifications were not performed.
HOLDING TIME (7 days extract; elutriate prep ≤ 14 days from collection)	Pesticides: Acceptable PCBs: Acceptable
STANDARD REFERENCE MATERIALS (SRM) SAMPLE(S)	SRM %Difference: (≤ 30%D of cv on avg for analytes > 10x MDL; ≤35% of all recoveries within a set of analytes > 30%D; No SRM recovery > 50%) Pesticides/PCBs: SRM samples were not analyzed. The LCS may be used if it is representative of marine water. There are no supporting data to indicate that the LCS matrix was marine water vs. laboratory grade water. Also, since the LCS analyzed does not include all of the required analytes it can not be used.
GENERAL QUALITY CONTROL SAMPLES (Replicate Samples; Matrix Spikes; Surrogates; Method Blanks)	Triplicate %RSD: (≤30% for analytes > 10x the MDL, < 35% RSDs can be >30%, No RSD > 70% for analytes > 10x MDL) Pesticides: Triplicate analysis was performed on sample E-SSH05-A. The %RSDs were not calculated. Triplicate analysis was not performed on sample E-SSH05-B. PCBs: Triplicate analysis was performed on sample E-SSH05-A. The %RSDs were not calculated. Triplicate analysis was not performed on sample E-SSH05-B. Matrix Spike % Recovery: (50 - 120% for at least 80% of the analytes) Pesticides: The MS sample does not include all of the required analytes. PCBs: The MS sample does not include all of the required analytes. Congeners 28, 52, 101, 118 were over recovered. Surrogate % Recovery: (30 - 150%) Pesticides: The recoveries are within the established criterion. PCBs: The recoveries are within the established criterion. Method Blank Results: (No analyte > 3x the target MDL) Pesticides: The results are within the established criterion. PCBs: The MRL reported are greater than 3X the target MDL specified in the RTM.
DATA ACCEPTABILITY Acceptable; Acceptable w/Condition; Unacceptable; Not Determined	Pesticides: Not Acceptable PCBs: Not Acceptable

METALS	TISSUE
MDL VERIFICATION STUDY(As 1.0; Cd 0.1; Cr 0.2; Cu 1.0; Pb 0.1; Hg 0.02; Ni 0.1; Ag 0.05; Zn 1.0, ug/g wet wt.)	MDL verifications were not performed with the project. The lab submitted past MDL studies for the parameters of interest in the matrix of interest. The MDL studies submitted were reviewed but could not be used since the results are reported in dry weight.
HOLDING TIME (28 days Hg; 6 mo. other metals; 1 year deep freeze)	Acceptable
STANDARD REFERENCE MATERIALS (SRM) SAMPLE(S)	SRM %Difference: (≤20%D of cv; ≤ 35% of all recoveries can be >20%D of cv; No recovery > 50%D) *M. nasuta*: Not Determined *N. virens:* Not Determined
GENERAL QUALITY CONTROL SAMPLES (Replicate Samples; Matrix Spikes; Method Blanks)	Triplicate %RSD: (≤20% for analytes > 10x MDL; ≤35% of RSDs > 20%; No RSD > 70% for analytes > 10x MDL) *M. nasuta*: Not Determined *N. virens*: Not Determined Matrix Spike % Recovery: (75 - 125%) *M. nasuta*: Not Determined *N. virens*: Not Determined Method Blank Results: (No analyte > 3x the target MDL) *M. nasuta*: Not Determined *N. virens*: Not Determined
DATA ACCEPTABILITY Acceptable; Acceptable w/Condition; Unacceptable; Not Determined	*M. nasuta*: Not Determined *N. virens*: Not Determined While reviewing metals in tissue data, it could not be determined from the information provided which samples, including the QC samples were analyzed in batches. Although run logs were obtained from the lab, the analytical batches with the associated QC samples could not be determined.

METALS	SITE WATER
MDL VERIFICATION STUDY	(Cd 0.025, Cr 1.0, Cu 0.3, Pb 0.3, Hg 0.002, Ni 0.3, Ag 0.1, Zn 0.15 ug/L) MDL verifications were not performed.
HOLDING TIME (Hg 28 days; 90 days for 1631E; 6mo. other metals)	Acceptable
STANDARD REFERENCE MATERIALS (SRM) SAMPLE(S)	SRM %Difference: (≤20%D of cv; ≤ 35% of all recoveries can be >20%D of cv; No recovery > 50%D) A SRM samples was not analyzed. The LCS may be used if it is representative of marine water. There are no supporting data to indicate that the LCS matrix was marine water vs. laboratory grade water. Zinc exceeds 50%D from the true value.
GENERAL QUALITY CONTROL SAMPLES (Replicate Samples; Matrix Spikes; Method Blanks)	Triplicate %RSD: (≤20% for analytes > 10x MDL; ≤35% of RSDs > 20%; No RSD > 70% for analytes > 10x MDL) Sample analysis was not performed in triplicate as required in the RTM. Matrix Spike % Recovery: (75 - 125%) MS was not performed correctly. It appears that the only analyte spiked was Hg. The remaining analytes were not included in the matrix spike sample. Method Blank Results: (No analyte > 3x the target MDL) Mercury is reported as not detected in the method blank, however both the MRL and MDL exceed 3x the target detection limit in the RTM. All remaining analytes are within the established criterion.
DATA ACCEPTABILITY Acceptable; Acceptable w/Condition; Unacceptable; Not Determined	Not Acceptable

METALS	ELUTRIATE **(RS-SJH05)**
MDL VERIFICATION STUDY	(Cd 0.025, Cr 1.0, Cu 0.3, Pb 0.3, Hg 0.002, Ni 0.3, Ag 0.1, Zn 0.15 ug/L) MDL verifications were not performed.
HOLDING TIME (Hg 28 days; 90 days for 1631E; 6mo. other metals)	Acceptable
STANDARD REFERENCE MATERIALS (SRM) SAMPLE(S)	SRM %Difference: (≤20%D of cv; ≤ 35% of all recoveries can be >20%D of cv; No recovery > 50%D) A SRM samples was not analyzed. The QCS may be used if it is representative of marine water. There are no supporting data to indicate that the QCS matrix was marine water vs. laboratory grade water.
GENERAL QUALITY CONTROL SAMPLES (Replicate Samples; Matrix Spikes; Method Blanks)	Triplicate %RSD: (≤20% for analytes > 10x MDL; ≤35% of RSDs > 20%; No RSD > 70% for analytes > 10x MDL) Sample analysis was not performed in triplicate as required in the RTM. Matrix Spike % Recovery: (75 - 125%) A matrix spike sample was not performed. Method Blank Results: (No analyte > 3x the target MDL) Mercury is reported as not detected in the method blank, however both the MRL and MDL exceed 3x the target detection limit in the RTM. All remaining analytes are within the established criterion.
DATA ACCEPTABILITY Acceptable; Acceptable w/Condition; Unacceptable; Not Determined	Not Acceptable.

METALS	ELUTRIATE **(E-SSH05-A & B)**
MDL VERIFICATION STUDY	(Cd 0.025, Cr 1.0, Cu 0.3, Pb 0.3, Hg 0.002, Ni 0.3, Ag 0.1, Zn 0.15 ug/L) MDL verifications were not performed.
HOLDING TIME (Hg 28 days; 90 days for 1631E; 6mo. other metals)	Acceptable
STANDARD REFERENCE MATERIALS (SRM) SAMPLE(S)	SRM %Difference: (≤20%D of cv; ≤ 35% of all recoveries can be >20%D of cv; No recovery > 50%D) A SRM samples was not analyzed. The LCS may be used if it is representative of marine water. There are no supporting data to indicate that the LCS matrix was marine water vs. laboratory grade water. Zinc exceeds 50%D from the true value.
GENERAL QUALITY CONTROL SAMPLES (Replicate Samples; Matrix Spikes; Method Blanks)	Triplicate %RSD: (≤20% for analytes > 10x MDL; ≤35% of RSDs > 20%; No RSD > 70% for analytes > 10x MDL) Triplicate analysis was performed on sample E-SSH05-A. The %RSDs were not calculated. Triplicate analysis was not performed on sample E-SSH05-B. Matrix Spike % Recovery: (75 - 125%) MS was not performed correctly. It appears that the only analyte spiked was Hg. The remaining analytes were not included in the matrix spike sample. Method Blank Results: (No analyte > 3x the target MDL) Mercury is reported as not detected in the method blank, however both the MRL and MDL exceed 3x the target detection limit in the RTM. All remaining analytes are within the established criterion.
DATA ACCEPTABILITY Acceptable; Acceptable w/Condition; Unacceptable; Not Determined	Not Acceptable